भारतीय मानक
Indian Standard

IS 8767 : 2020

रंग रोगन के लिए केलशियम कार्बोनेट, तलछट और सक्रिय — विशिष्टि

(पहला पुनरीक्षण)

Calcium Carbonate, Precipitated, and Activated, for Paints — Specification

(First Revision)

ICS 87.060.10

© BIS 2020

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

January 2020 **Price Group 5**

Raw Materials for Paint, Varnishes and Related Products Sectional Committee, CHD 21

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Raw Materials for Paints, Varnishes and Related Products Sectional Committee had been approved by the Chemical Division Council.

Calcium carbonate, in the form of a fine dry powder, is used as an extender in paint formulations. IS 63 Whiting for Paint and Putty (third revision) covers specification for whiting, a naturally occurring form of calcium carbonate used as an extender in paint industry an also meant for manufacture of putty. Precipitated and activated calcium carbonates in the chemically processed form are not so far covered in any Indian Standard for paints. In the light of recent experience in selection and use of extenders in paint industry and in view of chemically processed form of calcium carbonate coming into prominence for this purpose, the formulation of this standard was taken up by the concerned Committee to facilitate procurement of acceptable quality of precipitated and activated calcium carbonates for use as extender in paint industry.

The Committee responsible for the formulation of this standard was aware of the need for prescribing additional requirements for specific surface area and waterproofing characteristics for activated calcium carbonate. This was, however, not found possible in view of the fact that necessary testing facilities are not available commercially in the country at present. The matter would be reconsidered after such facilities become available.

In this revision two requirements namely, whiteness and water demand are included. Whiteness is important test as it has impact on final colour of paint. For coloured paints better whiteness of material gives clean shades. Water demand in otherwise flow point gives indication of particle size of the material and therefore is related to several properties like hiding, settling tendency and viscosity.

This standard contains **6.1** which call for agreement between the purchaser and the supplier.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CALCIUM CARBONATE, PRECIPITATED, AND ACTIVATED, FOR PAINTS — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for calcium carbonate, precipitated, and activated, intended for use as extender in paint industry.

2 REFERENCES

The standards listed in Annex A contains provisions which through reference in this text, constitute provisions of and necessary adjuncts to this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated in Annex A.

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 1303 and IS 33 shall apply.

4 TYPES

The material shall be of the following two types:

a) *Type 1* — Calcium carbonate, precipitated; and
b) *Type 2* — Calcium carbonate, activated.

5 REQUIREMENTS

5.1 The material shall be in the form of fine, white microcrystalline, free flowing powder or in such a condition that it can be reduced to the powder form by crushing without grinding action, under a palette knife. In addition, the activated calcium carbonate shall consist of precipitated calcium carbonate coated with a thin hydrophobic layer of fatty acids or their salts. The material shall be stable in air, free from visible impurities and shall closely resemble the approved sample in colour.

5.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Calcium Carbonate, Precipitated and Activated, for Paints

(*Clauses* 5.2 *and* 8)

Sl No.	Characteristic	Requirement		Method of Test, Annex	Ref to Clause No. in IS 33
(1)	(2)	(3)	(4)	(5)	(6)
		Type 1	Type 2		
i)	Volatile matter, percent by mass, *Max*	1.0	1.0	—	**8**
ii)	Residue on sieve, percent by mass, *Max*	0.25	0.25	—	**9**
iii)	Oil absorption[1]	← 30 to 80 →		—	**10**
iv)	Matter soluble in water (by cold extraction method), percent by mass, *Max*	0.3	0.3	—	**19**
v)	*p*H value	← 9 to 10.5 →		—	**21**
vi)	Bulk density	As agreed to between purchaser and the supplier		—	**17**
vii)	Particle size distribution, percent by mass, finer than 10 microns, *Min*	96	—	B	—
viii)	Loss on ignition, percent by mass	43.5 to 44.5	43 to 46	C	—
ix)	Insoluble siliceous matter, percent by mass, *Max*	0.2	0.2	D	—
x)	Calcium carbonate (as $CaCO_3$), percent by mass, *Min*	98	94	E	—
xi)	Total fatty matter, percent — by mass	2.8 to 3.2	F	—	
xii)	Water demand	120-140	120-130	G	—
xiii)	Whiteness :	— Comparable to Std —		H	—

[1] This shall, however, be within ± 10 percent of the approved sample, if any.

6 PACKING AND MARKING

6.1 Packing

The material shall be packed as agreed to between the purchaser and the supplier. The packing is subject to the provisions of the law in force in the country at that time.

6.2 Marking

6.2.1 The packages shall be securely closed and marked with the following particulars:

a) Name and type of the material;

b) Name of the manufacturer or his recognized trade-mark, if any;

c) Volume of the material;

d) Batch number or lot number in code or otherwise; and

e) Month and year of manufacture.

6.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6.3 Other details of packing and marking shall be in accordance with the instructions given by the purchaser.

7 SAMPLING

7.1 Preparation of Test Samples

Representative samples of the material shall be drawn as prescribed under **4** of IS 33.

7.2 Criteria for Conformity

A lot shall be declared as conforming to the requirements of this standard, if test results of the composite sample satisfy the requirements prescribed under **5**.

8 TESTS

Tests shall be conducted as prescribed in Annex B to H (Methods of test for precipitated and activated calcium carbonate for paints) and IS 33. Reference to relevant clauses of Annex A and IS 33 are given in col 5 and 6 of Table 1 respectively.

8.1 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
33 : 1992	Methods of sampling and test for inorganic pigments and extenders for paints (*third revision*)
63 : 2006	Whiting for paint and putty (*third revision*)
170 : 2004	Acetone — Specification (*fourth revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
266 : 1993	Sulphuric acid (*third revision*)
336 : 1973	Ether — Specification (*second revision*)
917 : 1976	Specification for activated calcium carbonate for rubber industry
918 : 1985	Specification for calcium carbonate, precipitated, for cosmetic industry
1303 : 1983	Glossary of terms relating to paints (*second revision*)
1070 : 1992	Reagent grade water (*third revision*)
10332 : 1982	Hydrofluoric acid, aqueous — Specification

ANNEX B

(*Table* 1 and *Clause* 8)

DETERMINATION OF PARTICLE SIZE DISTRIBUTION

B-1 GENERAL

Because of the ease of execution and good reproducibility the Andreasen method is included as the referee method. Other methods may, however, be used by agreement between the concerned parties, but in such cases it will be necessary also to agree on appropriate limits.

B-2 PRINCIPLE

The rate of fall of spherical particles through a medium in which they are dispersed is proportional to the square of the particle diameter (Stokes' law). The Andreasen method for determination of particle size distribution makes use of this relationship and expressed the particle size distribution in terms of distribution of spherical particles of the same settlement rate.

B-3 OUTLINE OF THE METHOD

In this determination, a dilute suspension is prepared and the concentration of solids at a fixed point below the surface is determined at a series of time intervals calculated to correspond to certain equivalent spherical diameters.

B-4 APPARATUS

See Fig. 1.

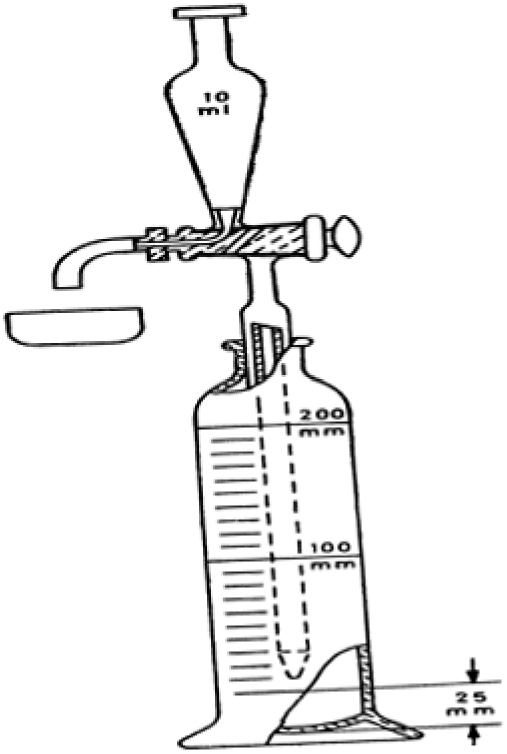

FIG. 1 SEDIMENTATION VESSEL AND PIPETTE

B-4.1 Sedimentation Vessel — Glass about 56 mm internal diameter and having a graduated scale from 0 to 200 mm marked on its side. The zero graduation line shall not be less than 25 mm from the inside base of the vessel and the capacity of the vessel up to the 200 mm mark shall be about 550 to 620 ml.

B-4.2 Pipette — Fitted with a two-way tap and side discharge tube. The capacity of the pipette to the graduation line is conveniently 10 ml. A bell-shaped dome with a ground glass joint to fit the neck of the sedimentation vessel is fused to the pipette. A small vent hole is made in this dome. The inlet to the pipette stem shall be level with the zero line on the sedimentation vessel. The stem from the pipette bulb to the sampling inlet shall be constructed of capillary glass tubing with a bore not less than 1 mm and not more than 1.3 mm. The tube above the bulb shall be 4 to 4.5 mm bore.

B-4.3 Constant-Temperature Bath — Transparent-sided of at least 15-litres capacity, maintained at a temperature of 27 ± 0.5°C, into which the sedimentation vessel can be immersed up to the 200 mm graduation mark. The bath shall be positioned away from sources of vibration.

B-4.4 Mechanical Stirrer — Capable of rotating at a suitable speed for complete dispersion (1 000 ± 100 rpm is generally suitable). The stirrer shall be designed to lift the dispersion and to avoid the creation of a vertex.

NOTE — A suitable stirrer may be made from an approximately 40 mm diameter brass disc with four equally spaced cuts, the cut sections being turned upwards at an angle of 30° to the horizontal.

B-4.5 Dispersion Vessel — Of appropriate dimensions, such as a 1 000 ml gas jar.

B- 4.6 Weighing Balance — Having least count 0.1 mg and suitable range.

B-4.7 Drying Oven — Capable of being maintained within a temperature range suitable for evaporation of the suspending liquid, for example for water range is 105 ± 2°C and least count 0.5°C.

B-4.8 Weighing Bottles —Wide-mouthed, suitable for evaporation, of capacity not less than 20 ml, or a small laboratory centrifuge and centrifuge tubes preferably of 20-ml capacity but of not less than 10 ml capacity.

B-4.9 Stop-Watch or Stop-Clock

B-5 PREPARATION FOR THE TEST

B-5.1 Calibration of Pipette — Thoroughly clean the pipette. Partly fill the sedimentation vessel with water. Set the top in the sampling position and by means of a rubber tube suck water into the bulb to the level of the graduation line. Reverse the tap to the discharge position and allow the water to drain into a tared weighing bottle. Apply pressure through the rubber tube to blow any water remaining in the bulb and the discharge tube into the weighing bottle. Weigh the bottle to the nearest 0.001 g and calculate from this mass the internal volume Vp of the pipette.

B-5.2 Calibration of Sedimentation Vessel — Thoroughly clean the sedimentation vessel, weigh it, with the pipette in place, to the nearest 0.1 g. Then fill it to the 200 mm mark with water at 27 ± 0.5°C and reweigh. Calculate from the mass of water the internal volume Vs of the vessel.

B-5.3 Sample for Analysis — Using 3.5 g of accurately weighed material with 0.07 to 0.14 g of suitable dispersing materials (*see* Note) prepare a dispersion by placing the test portion in the dispersion vessel adding dispersion solution at 27 ± 0.5°C to give a total volume of about 500 ml and stirring with the mechanical stirrer for 15 min. Immediately pour all the suspension into the sedimentation vessel and make up to the 200 mm line with water or aqueous alcohol as appropriate. Transfer the sedimentation vessel to the constant temperature bath.

NOTE — Certain sodium salts of polymethacrylates have been found most suitable. Such materials are sold as polysalts, dispex, etc.

B-6 PROCEDURE

B-6.1 Sedimentation

Allow the sedimentation vessel to stand immersed to the 200 mm mark until it has reached the temperature of the bath. Record this temperature, which shall not differ greatly from that of the room. When temperature equilibrium has been attained, mix the contents thoroughly by placing a finger over the vent hole and inverting the vessel several times. Immediately after mixing, replace the vessel and start the stop-clock or stop-watch.

B-6.2 Withdraw from the suspension by means of the pipette a series mixing, starting at time t_1 corresponding to a particle size of about 20 µm (calculated in accordance with **B-1.4.3**) and thereafter in progression so that the limiting diameters stand in a √2 : 1 progression. Alternatively, the samples may be taken at convenient times approximately in a 2:1 progression and the exact spherical particle diameters corresponding to each sample calculated. The time to fill the pipette shall be about 20 s.

B-6.3 Discharge the suspension from the pipette into a tared weighing bottle. When the bulb has drained, remove the rubber suction tube and run 5 to 7 ml of water from an ordinary 10 ml pipette into the bulb of the sedimentation pipette to wash into the weighing bottle any particles adhering to the surface. The stem of the sedimentation pipette shall remain filled with suspension. Dry the contents of the weighing bottle at 105°C until the difference between successive weighing is not greater than 0.1 mg. Calculate the mass of the fraction m_n allowing for the mass of dispersing agent, which shall be determined by carrying out a blank test. The determination of different fractions may be carried out by means of chemical methods if that procedure is more suitable.

B-6.4 The depth of immersion of the pipette decreases as each fraction is withdrawn. Determine the exact decrease in depth by experiment and allow for it in the subsequent calculation. Suppose that the decrease is 4 mm for each fraction withdrawn, and that initially the depth of immersion of the pipette was 200 mm, then the depth after the first fraction has been withdrawn will be 196 mm, and the mean depth h_1 used for calculation of the initial diameter corresponding to the first fraction will be 198 mm. The mean depth h_2 for the second fraction will be 194 mm, and so on.

B-6.5 Calculation of Withdrawal Time

The withdrawal time, t_n, in seconds, for the *n*th sample is given by the equation:

$$t_n = \frac{18\eta h_n}{(\rho 2 - \rho 1)\, g\, d_n^2} \times 10^6$$

Where,

η = absolute viscosity, of the medium in newton seconds per square metre;

h_n = depth, , at which the *n*th sample extraction is made in millimetres;

$\rho 1$ = density, of the medium in megagrams per cubic metre;

$\rho 2$ = density, of the particle in megagrams per cubic metre (or g/cm^3);

g = acceleration due to gravity, in metres per second square; and

d_n = limiting stokes diameter, , corresponding to the *n*th sample extraction, in micrometres.

B-7 Calculation of Cumulative Percentage Undersize

The cumulative percentage by mass, P_n, of particles smaller than each of the limiting stokes diameter d_n for each time interval t_n is given by the formula:

$$P_n = \frac{m_n \times V_s}{m_s \times V_p} \times 100$$

where,

m_n = mass of the fraction corrected for the mass of the dispersing agent, in g;

V_s = volume of the sedimentation vessel, in ml;

m_s = mass of the test portion, in g; and

V_p = volume of the pipette, in ml.

B-8 Repetition of Test

Repeat the procedure on a further amount of suspension prepared from the same sample. The results of the test shall be accepted only if the percentages by mass, less than the same limiting stokes diameter, do not differ by more than 4 percent.

B-9 Expression of Results

Plot the results of the analysis with the micrometric sizes as abscissae and the percentages undersize as ordinates. From the smooth curve drawn through the points, select the cumulative percentages corresponding to the series required. Repeat the results to the nearest 1 percent.

ANNEX C

(*Table* 1 *and Clause* 8)

DETERMINATION OF LOSS ON IGNITION

C-1 APPARATUS

C-1.1 Weighing Balance

Having least count 0.1 mg and of suitable range.

C-1.2 Platinum Crucible

Of suitable capacity

C-1.3 Desiccator

C-1.4 Muffle Furnace

Having maximum range not less than 1 200°C and least count 5°C

C-1.5 Drying Oven

Capable of being maintained within a temperature range suitable for evaporation of the suspending liquid, for example, for water range is 105 ± 2°C and least count 0.5°C.

C-2 PROCEDURE

Weigh accurately about 1 g of the test sample, previously dried at 100 ± 2°C to constant mass (M_1) into a platinum crucible. Heat the crucible, along with its contents at a temperature of 1 000 ± 25°C in a muffle furnace and ignite for 2 h, cool in a desiccator containing phosphorus pentoxide and weigh. Repeat heating, cooling and weighing till constant mass (M_2) is obtained.

NOTE — Care should be taken to avoid hydration of the quicklime formed.

C-3 CALCULATION

Express the loss in mass as the percentage of material taken for the test is given by the formula:

$$\text{Loss in mass } \textit{(percent)} = \frac{M_1 - M_2 \times 100}{M_1}$$

ANNEX D

(*Table* 1 and *Clause* 8)

DETERMINATION OF INSOLUBLE SILICEOUS MATTER

D-1 APPARATUS

D-1.1 Weighing Balance

Having least count 0.1 mg and of suitable range.

D-1.2 Platinum Crucible

Of suitable capacity.

D-1.3 Desiccator

D-1.4 Muffle Furnace

Having maximum range not less than 1 200°C and least count 5°C.

D-1.5 Drying Oven

Capable of being maintained within a temperature range suitable for evaporation of the suspending liquid for example, for water range is 105 ± 2°C and least count 0.5°C.

D-2 REAGENTS

D-2.1 Concentrated Hydrochloric Acid (*see* IS 265)

D-2.2 Concentrated Sulphuric Acid (*see* IS 266)

D-2.3 Hydrofluoric Acid

Approximately 40 percent by volume *(see* IS 10332).

D-3 PROCEDURE

Weigh accurately about 5 g of previously dried test sample and ignite as in A-**2.1**. Add slowly concentrated hydrochloric acid to dissolve the material. Dilute it to 300 ml, allow to stand for 10 min and then filter. Wash the residue with water until free from chlorides (when tested with silver nitrate solution, the washings should not turn milky). Ignite the residue with filter paper in a platinum crucible. Cool the crucible, add two drops of concentrated sulphuric acid and then heat to constant mass. Cool in a desiccator and weigh. Add to this, one or two drops of concentrated sulphuric acid and 6 to 7 ml of hydrofluoric acid in two installments. Dry and then ignite at a temperature of about 850°C for 2 h, cool it in a desiccator and weigh. Repeat heating, cooling and weighing till constant mass is obtained.

D-4 CALCULATION

Insoluble siliceous matter, percent by mass =

$$\frac{100\,(A - B)}{m}$$

where,

A = mass of the residue after treatment with concentrated sulphuric acid, in g;

B = mass of the residue after treatment with concentrated sulphuric acid and hydrofluoric acid, in g; and

m = mass of the sample taken for test, in g.

ANNEX E

(*Table* 1 *and Clause* 8)

DETERMINATION OF CALCIUM CARBONATE

E-1 GENERAL

Two methods have been prescribed for this test, namely, Method A and Method B. Method A is the classical method and Method B is the complexometric method. In case of dispute Method A shall be followed:

E-2 METHOD A

E-2.1 Outline of the Method

Calcium is precipitated as its oxalate and dissolved in dilute sulphuric acid and the solution is titrated against permanganate solution.

E-2.2 Apparatus

E-2.2.1 *Weighing Balance* — Having least count 0.1 mg and of suitable range.

E-2.2.2 *Platinum Crucible* — Of suitable capacity

E-2.2.3 *Desiccator*

E-2.2.4 *Glasswares* — Like beaker, graduated flask, pipette, etc.

E-2.2.5 *Drying Oven* — Capable of being maintained within a temperature range suitable for evaporation of the suspending liquid, for example, for water range is 105 ± 2°C and least count 0.5°C.

E-2.3 Reagents

E-2.3.1 *Dilute Hydrochloric Acid* — Approximately 5 N (*see* IS 265).

E-2.3.2 *Ammonium Oxalate Solution* — Saturated.

E-2.3.3 *Dilute Ammonium Hydroxide* — Approximately 5 N and 1 N.

E-2.3.4 *Dilute Potassium Permanganate Solution* — 0.01N (approximately).

D-2.3.5 *Dilute Sulphuric Acid* — Approximately 5 N (*see* IS 266)

E-2.3.6 *Standard Potassium Permanganate Solution* — 0.1 N.

E-2.4 Prepared Sample Solution

Weigh accurately about 1 g of sample, previously dried at 100 ± 2°C, into a platinum crucible and ignite as in C-**1**. Transfer the contents of the crucible completely to a 250 ml beaker with a jet of water from a wash bottle. Rinse the crucible with a few millilitres of dilute hydrochloric acid to detach particles adhering to the crucible. Continue addition of acid to the beaker until a total of 40 ml of acid has been added. Heat the solution and boil for about 10 minutes. Cool and transfer completely to a 200 ml graduated flask, add water to make up the volume and mix thoroughly.

E-2.5 Procedure

Pipette out 50 ml of the prepared sample solution into a beaker and add 75 ml of water. Heat almost to boiling and add 15 ml of ammonium oxalate solution. To the hot solution, constantly stirred, add dilute ammonium hydroxide (5 N) dropwise until the mixture is strongly alkaline. Let it stand for 2 h. Decant the supernatant liquid through a filter paper. Transfer all the precipitate from beaker to the filter paper by addition of cold water. When all the precipitate has been transferred, wash the precipitate in the filter paper first with dilute ammonium hydroxide (1 N) and then with cold water until the filtrate does not decolourize dilute potassium permanganate solution in presence of dilute sulphuric acid. Puncture the filter paper and transfer the precipitate to a beaker with a fine jet of hot water. Add about 30 ml of dilute sulphuric acid, heat at 60°C and titrate with standard potassium permanganate solution. Towards the end of the titration introduce the filter paper which was used for filtration into the titration vessel and carry out the titration till the end point is reached. Carry out a blank test with all reagents, following the procedure for the test but without the material:

E-2.6 Calculation

$$\text{Calcium carbonate (as } CaCO_3\text{), percent by mass} = \frac{20.02\,(V_1 - V_2)\,N}{M}$$

where,

V_1 = volume of standard potassium permanganate solution used for the test, in ml;

V_2 = volume of standard potassium permanganate solution used for the blank test, in ml;

N = normality of standard potassium permanganate solution; and

M = mass of the material taken for the test, in g.

E-3 METHOD B

E-3.1 Outline of the Method

E-3.2 Apparatus

The solution of the material, after adjustment of *p*H, is titrated with EDTA solution using murexide indicator.

E-3.2.1 *Weighing Balance* — Having least count 0.1 mg and of suitable range.

E-3.2.2 *Platinum Crucible* — Of suitable capacity.

E-3.2.3 *Desiccator*

E-3.2.4 *Glasswares* — Like beaker, graduated flask, pipette, etc.

E-3.2.5 *Drying Oven* — Capable of being maintained within a temperature range suitable for evaporation of the suspending liquid e.g. for water range is 105 ± 2°C and least count 0.5°C.

E-3.3 Reagents

E-3.3.1 *Dilute Hydrochloric Acid* — Approximately 5 N.

E-3.3.2 *Dilute Sodium Hydroxide Solution* — Approximately 1 N.

E-3.3.3 *Standard Calcium Carbonate Solution* — 0.01 N. Weigh 0.500 4 g of calcium carbonate, dried at 110°C, and dissolve in the minimum quantity of dilute hydrochloric acid. Make up the volume with later to 1 litre.

E-3.3.4 *Murexide Indicator* — Triturate 0.2 g of murexide with 50 g of potassium chloride until a homogeneous mixture is obtained.

E-3.3.5 *Standard Disodium Ethylenediamine Tetra-acetate (EDTA) Solution* — Weigh 3.7 g of disodium ethylenediamine tetra-acetate dihydrate and dissolve in water. Make up the volume to 1 litre. Standardize the solution against standard calcium carbonate solution as under **E-2.3.3**.

E-3.4 Prepared Sample Solution

Weigh accurately about 3 g of previously dried test sample into a platinum crucible and ignite as in **C-1**. Transfer the contents of the crucible completely to a 250 ml beaker with a jet of water from a wash bottle. Rinse the crucible with a few millilitres of dilute hydrochloric acid to detach particles adhering to the crucible. Continue addition of acid to the beaker until a total of 30 ml has been added. Heat the solution and boil for about 10 minutes. Cool and transfer quantitatively to a 500 ml graduated flask. Pipette out 100 ml of this solution and dilute further to 1 litre. Use this diluted solution for test as under **E-2.5**.

E-3.5 Procedure

Pipette out 10 ml of the prepared sample solution into a 250 ml conical flask. Add 25 ml of water and 10 ml of dilute sodium hydroxide solution (*p*H of the mixture should be more than 12). Add 0.3 g of murexide indicator and titrate with standard EDTA solution until the colour changes from red to distinct blue violet.

E-3.6 Calculation

$$\text{Calcium carbonate (as CaCO}_3\text{), percent by mass} = \frac{2.502\, VN}{M}$$

Where,

V = volume of standard EDTA solution, in ml;

N = normality of standard ETDA solution; and

M = mass of the material taken for the test, in g (*see* **D-4.2.2)**.

ANNEX F

(*Table* 1 and *Clause* 8)

DETERMINATION OF TOTAL FATTY MATTER

F-1 APPARATUS

F-1.1 Weighing Balance—Having least count 0.1 mg and of suitable range.

F-1.2 Seperating Funnel

F-1.3 Desiccator

F-1.4 Glasswares — Like beaker, graduated flask, pipette conical flask etc.

F-2 REAGENTS

F-2.1 Ether —*See* IS 336.

F-2.2 Hydrochloric Acid —Approximately 5 N (*see* IS 265).

F-2.3 Acetone —*See* IS 170

F-3 PROCEDURE

Weigh accurately about 5 g of the material in a 400 ml beaker and wet the material by adding a little ether. Cover with a cover-glass and carefully add (a few millilitres at a time) 50 ml of hydrochloric acid. Boil, dilute to about 100 ml with water and cool to room temperature. Transfer to a 300 ml separating funnel, rinse out the beaker with 25 ml of ether and transfer to the separating funnel. Shake gently, release the pressure by inverting the funnel and gradually open the tap. Allow the two layers to separate completely, then run off the aqueous layer into the original beaker. Filter the ether layer through a dry filter paper into a tared conical flask. Transfer the aqueous layer back into the separating funnel and extract twice as before but using 15 ml portions of ether. Filter the combined ether extracts also through the same filter paper and collect the filtrate in the tared conical flask. Finally wash the filter paper with ether, distil the ether until only 5 ml remains in the flask, then add about 5 ml of acetone and evaporate the mixture on a water bath. Dry the residue at 60°C. In order to reduce the drying time to about 15 minutes and minimize volatilization and oxidation of fatty acid, blow through the flask a gentle current of an inert gas two or three times during drying. Cool the flask in a desiccator and reweigh.

F-4 CALCULATION

$$\text{Total fatty matter, percent by mass} = \frac{100\,(A - B)}{M}$$

where ,

A = mass of the conical flask containing the dried residue, in g;

B = mass of the conical flask, in g; and

M = mass of the material taken for test, in g.

ANNEX G

(*Table* 1 *and Clause* 8)

DETERMINATION OF WATER DEMAND

G-1 GENERAL

There are two different methods applicable for the estimation of water. One for natural and another for synthetic calcium carbonate (precipitated and activated calcium carbonate).

G-2 METHODOF TEST FOR PRECIPITATED AND ACTIVATED CALCIUM CARBONATE

G-2.1 This method is applicable for all types of synthetically prepared as well as post treated calcium carbonate which are not fully hydrophilic in nature.

G-2.2 Apparatus

G-2.2.1 *Weighing Balance* — Having least count 0.1 mg and of suitable range.

G-2.2.2 *Desiccator*

G–2.2.3 Glasswares — Like beaker, pipette, burette, conical flask, etc.

G-2.3 Reagents

G-2.3.1 *Aqueous Solution,* of polyacrylate based low foaming anionic surfactant

G-2.3.2 *Hydrochloric Acid,* approximately 5 N (*see* IS 265).

G-2.3.3 *Acetone, see* IS 170.

G-2.4 Procedure

G-2.4.1 Weigh 20 g of the sample in 150 ml glass beaker. Add 5 ml of 10 percent aqueous solution of polyacrylate based low foaming anionic surfactant (for example Indofil 731 ex M/s Indofil India or Lutron 731 ex M/s Rohm and Haas) and mix thoroughly. Add distilled water using burette, in small installments of 1 ml each with constant stirring with a glass rod, till the free flowing powder is converted into a paste form. Tilt the beaker carefully at 45° angle to allow the paste to flow. At this point, note the burette reading.

G- 2.4.2 To obtain the accurate results, repeat the test with the same sample as indicated in step **G-3.1** above till the last but one ml and later on adding distilled water drop by drop till the end point that is the flow point.

G-3 CALCULATIONS

Water demand point for the sample =

$$\frac{\text{Burette reading} + 5}{\text{Weight. of sample taken for the test}} \times 100$$

Report the water demand as ml of water per 100 g powder.

ANNEX H

(*Table* 1 *and Clause* 8)

DETERMINATION OF WHITENESS OF EXTENDERS

H-1 GENERAL

This method is applicable for the estimation of whiteness of extenders used in paint preparation.

H-2 APPARATUS

H-2.1 Glass Plates, of size 250 mm × 100 mm of thickness 3 to 4 mm,

H-2.2 Spatula and Sharp Edged Knife

H-3 CHEMICAL

H-3.1 Distilled Water (*see* IS 1070).

H-3.2 Mineral Turpentine.

H-4 PROCEDURE

H-4.1 Clean the glass plate. Place a small amount of the extender sample on the glass plate. Carefully flatten the heap using a clean dry spatula and press such that a dry cake is formed. Cut off the left and right edges of the cake to get a rectangular block (Now place another sample near to the previous sample without leaving any space in between. Carefully flatten the heap using a clean dry spatula and press such that a dry cake is formed. Cut off the left and right edges of the cake to get a rectangular block. Continue in the same manner for all the samples of the extender). Now keep the standard sample near to the test sample without leaving space in between and flatten it carefully so that both heaps are touching other. Press the heap to form a dry cake. Carefully wet both heaps using water or mineral turpentine, as applicable and drain out excess of water or mineral turpentine by holding the plate in inclined manner.

NOTE — Use water for hydrophilic extenders like clays and mineral turpentine for hydrophobic extenders like talc silica, calcium carbonate.

H-4.2 Observe the colour of the sample against that of standard.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: CHD 21 (1859).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	2323 7617 2323 3841
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi KOLKATA 700054	2337 8499, 2337 8561 2337 8626, 2337 9120
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg CHANDIGARH 160019	265 0206 265 0290
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	2254 1216, 2254 1442 2254 2519, 2254 2315
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093	2832 9295, 2832 7858 2832 7891, 2832 7892

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE. DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi